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(54) **Liquid fabric conditioner and dryer sheet fabric conditioner containing fabric softener, aminosilicone and bronsted acid compatibiliser**

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EP 0 459 821 B1

Description**BACKGROUND OF THE INVENTION**5 **FIELD OF THE INVENTION**

The instant invention relates in a first aspect to conditioning of fabrics in an aqueous wash bath, to liquid compositions containing fabric conditioning ingredients and to processes for making the compositions. In a second aspect the invention relates to fabrics in tumble-dryer automatic dryers. More particularly,
10 it relates to an article in the form of a flexible substrate carrying a fabric conditioning composition.

RELATED ART

Silicones have been applied to fabrics during manufacture of fabrics or during the make up of articles of
15 clothing. Mixtures of quaternary ammonium salts and silicones have been described in the preparation of yarn and/or thread as described in EP-A-0 063 311 and as a means of suds control during the washing process as described in GB-A-2 185 752.

With respect to application of silicones to fabrics during a laundry process, GB-A-1,549,480; Burmeister et al., US-A-4,818,242; König et al., US-A-4,724,089; König et al., US-A-4,806,255; Dekker et al., US-A-
20 4,661,267 and Trinh et al., US-A-4,661,269 describe aqueous dispersions or emulsion of certain silicones of limited viscosity incorporated in liquid rinse-cycle fabric softening compositions. A fabric softening composition containing emulsified silicone is taught by Barrat et al in US-A-4,446,033. The rinse compositions taught by the '089, '255, '267 and '269 patents contain cyclic amine fabric softening agents and employ water-soluble Bronsted acids to control the pH of the aqueous compositions for proper dispersion of the
25 amine.

The compositions disclosed in the art contain individual particles of a silicone and individual particles of a fabric softening agent.

Chang, US-A-4,789,491 discloses fabric softening compositions containing quaternised di-esters or di-isopropanol amines. The compositions may optionally contain various types of silicone components.

30 Wells, US-A-4,308,024 discloses non-silicone fabric softening compositions consisting essentially of a water-insoluble cationic detergent surfactant and a C₈-C₂₄ alkyl- or alkenyl monocarboxylic acid.

The application of fabric softeners to fabrics in the tumble dryer by use of a flexible substrate carrying the fabric softeners is known in the art. The advantages of dryer added fabric conditioning include a more convenient time of addition in the laundry process and avoidance of undesirable interaction of softening
35 agents with detergents. Rudy et al., US-A-3,972,131 discloses dryer sheets including a silicone oil as an ironing aid. Kasprzak et al., US-A-4,767,548 discloses the use of certain silicones in dryer sheet formulations.

Coffindaffer et al, US-A-4,800,026 discloses curable amine functional silicones in fabric care compositions.

40 Japanese Patent Application 62/78,277 discloses chemically combined condensation products of amino modified silicone oils as softeners.

In the manufacture of the dryer added fabric conditioning sheets described in some of the references mentioned above, when silicones are mixed with fabric softeners, the resulting mixtures are non-homogeneous and phase separation occurs readily. The homogeneity of such mixtures is ensured only by
45 continuous vigorous agitation. An additional problem associated with the use of a nonhomogeneous mixture is the separation of actives at the point of application of the active mixture on the substrate resulting in unevenly impregnated sheets.

Bronsted acids described herein compatibilise aminosilicones with fabric softening agents. In the present invention the dispersed particle is a composite particle containing a mutually soluble mixture of an
50 aminosilicone, a fabric softening component and a Bronsted acid.

Critically, the aminosilicones in the composite particles of the present invention do not separate from the fabric softening agent during processing, on standing, during coating or solidifying on the dryer sheets. An additional advantage afforded by the present invention is a simplified manufacture of fabric conditioning liquids since silicone no longer has to be dispersed separately and can be introduced into the composition
55 simultaneously with a fabric softener.

Accordingly, it is a first object of the present invention to provide a liquid fabric conditioning composition which contains composite particles of a compatible mixture of a fabric softening component, an aminosilicone and a Bronsted acid.

It is a further object of the invention to provide processes by which the aforementioned composition can be manufactured.

Accordingly, it is a further object of the present invention to provide an article which provides for release of a fabric conditioning composition within an automatic laundry dryer, the composition containing a compatible mixture of a fabric softening component, an aminosilicone and a Bronsted acid.

These and other objects and advantages will appear as the description proceeds.

SUMMARY OF THE INVENTION

The present invention is based, in part, on the discovery that specific Bronsted acids are capable of compatibilising aminosilicones with certain conventional fabric softening agents. As a result of the use of Bronsted acids as described herein mutually compatible mixtures containing an aminosilicone and a fabric softening component comprising a cationic quaternary ammonium salt can be formed.

The present invention relates to discrete composite particles characterised in that they consist of a mutually compatible mixture of:

- a) at least 1 wt% of a fabric softening component comprising a cationic quaternary ammonium salt,
 - b) an amine functional organosilicone,
 - c) a Bronsted acid containing an alkyl group having at least 6 carbon atoms,
- the weight ratio of (c) : [(a) + (b)] being such that the mixture when in liquefied form is homogeneous.

A further aspect of the invention is a liquid fabric conditioning composition as claimed in claim 8 and an article for conditioning fabrics as claimed in claim 11.

Mutual compatibility as taught herein is critical and is ascertained by the appearance of the mixture containing an aminosilicone, a fabric softener and a Bronsted acid. When an aminosilicone, a fabric softener and a Bronsted acid are heated and mixed together, the resulting liquid mixtures are either transparent or opaque. In the transparent mixtures, Bronsted acid compatibilises aminosilicone with fabric softener and a mutually soluble mixture containing an aminosilicone, a fabric softener and a Bronsted acid is formed. Accordingly, transparent mixtures are suitable for use in the present invention. In the opaque mixtures an aminosilicone, a fabric softener and a Bronsted acid are not mutually soluble. These opaque mixtures are sometimes sufficiently stable for use as a coating for dryer sheet application.

Thus, the class of compatible mixtures as defined herein includes mutually soluble mixtures of an aminosilicone, a fabric softener and a Bronsted acid as well as mixtures wherein an aminosilicone, a fabric softener and a Bronsted acid form mutually stable dispersions. Compatibility of the mixture is critical and is determined by the Compatibility Test described below.

In its broadest aspect, some objects of the invention are accomplished by a liquid fabric conditioning composition which includes about 1% to about 60% of composite particles containing a mutually compatible mixture of a fabric softening component, an aminosilicone and a Bronsted acid. Of course, these particles can also be added to a liquid containing other fabric treating ingredients including, for example, softeners. Other objects of the invention are accomplished by an article comprising a flexible substrate carrying an effective amount of a fabric conditioning composition affixed thereto in a manner which provides for release of the conditioning composition within an automatic tumble dryer at dryer operating temperatures.

The fabric softening component employed herein may be any commonly used fabric softening agent complying with the above conditions provided that it includes at least a portion of cationic quaternary ammonium salts used singly or, optionally, in admixture with other softening agents such as nonionic softeners selected from the group of tertiary amines having at least one C₈₋₃₀ alkyl chain, esters of polyhydric alcohols, fatty alcohols, ethoxylated fatty alcohols, alkylphenols, ethoxylated alkylphenols, ethoxylated fatty amines, ethoxylated monoglycerides, ethoxylated diglycerides, mineral oils, polyols, carboxylic acids having at least 8 carbon atoms, and mixtures thereof.

The fabric conditioning compositions of the present invention include an organosilicone having an amine functionality, i.e. an aminosilicone.

The compositions also contain Bronsted acids which compatibilise an aminosilicone with a fabric softening component. Bronsted acids employed in the present invention have an alkyl group having at least 6 carbon atoms.

A certain amount of Bronsted acid is necessary to compatibilise an aminosilicate with a fabric softener. The weight ratio of the Bronsted acid to the combined weight of the aminosilicone and the fabric softening component is at least such that a mutually compatible and, preferably, transparent mixture of a fabric softening component, an aminosilicone and a Bronsted acid is formed, as determined by the Compatibility Test.

Each component of the present compositions: the fabric softening component, the aminosilicone and the Bronsted acid may provide fabric conditioning benefits including softness, fluffiness, static control, and other benefits when fabrics are comingled with compositions of the invention in an aqueous bath or in a tumble dryer.

- 5 Liquid conditioning compositions of the present invention include a liquid carrier and may be formulated as diluted or concentrated products.

DETAILED DESCRIPTION OF THE INVENTION

- 10 The liquid fabric conditioning composition of the present invention includes a cationic quaternary ammonium salt. The counterion is methylsulfate or any halide.

Examples of cationic quaternary ammonium salts include, but are not limited to:

1. Acyclic quaternary ammonium salts having at least two C₈ to C₃₀, preferably C₁₂ to C₂₂ alkyl chains, such as: ditallowdimethyl ammonium chloride, di(hydrogenated tallow)dimethyl ammonium chloride, distearyldimethyl ammonium chloride, dicocodimethyl ammonium chloride and the like;
- 15 2. Cyclic quaternary ammonium salts of the imidazolinium type such as di(hydrogenated tallow)dimethyl imidazolinium methyl sulfate, 1-ethylene-bis(2-tallow-1-methyl) imidazolinium methyl sulfate and the like;
3. Diamido quaternary ammonium salts such as: methyl-bis(hydrogenated tallow amidoethyl)-2-hydroxyethyl ammonium methyl sulfate, methyl bis(tallowamidoethyl)-2-hydroxypropyl ammonium methyl sulfate and the like;
- 20 4. Biodegradable quaternary ammonium salts such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium chloride, and N,N-di(tallowoyl-oxy-propyl)-N,N-dimethyl ammonium chloride and the like. When fabric conditioning compositions employ biodegradable quaternary ammonium salts, the pH of the composition is preferably adjusted to between about 2 and about 5. Biodegradable quaternary ammonium salts are described, for example, in U.S. Patents 4,137,180, 4,767,547 and 4,789,491 incorporated by reference herein.
- 25 5. Mixtures of water-insoluble cationic fabric softener and a polyalkoxylated ammonium salt as described in US-A-4,422,949. Such mixtures may be particularly suitable for incorporation in a concentrated form of the liquid compositions herein.

- 30 The fabric softening component may include other fabric softeners in addition to the cationic quaternary ammonium salts. Additional fabric softeners suitable for use herein can be selected from the following classes of compounds:

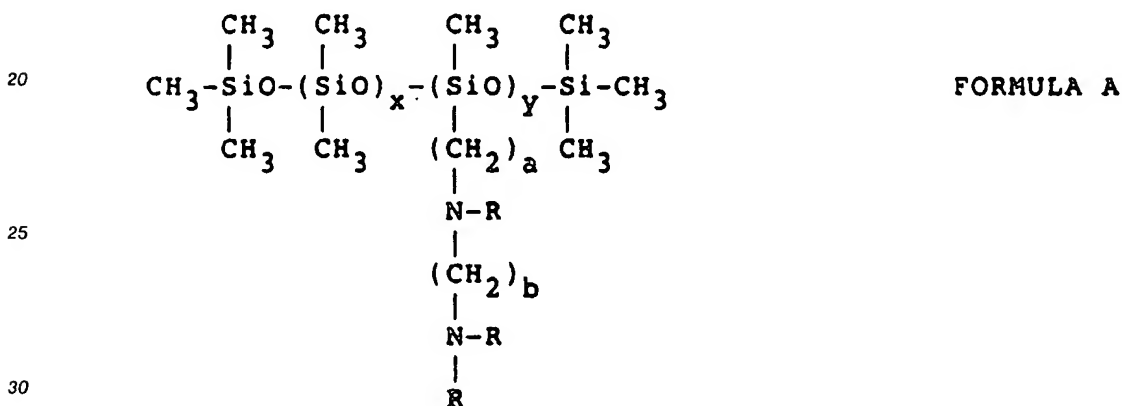
- i. Tertiary fatty amines having at least one and preferably two C₈ to C₃₀, preferably C₁₂ to C₂₂ alkyl chains. Examples include hardened tallow amine and cyclic amines such as 1-(hydrogenated tallow)-amidoethyl-2-(hydrogenated tallow) imidazoline. Cyclic amines which may be employed for the compositions herein are described in US-A-4,806,255 incorporated by reference herein.
- 35 ii. Carboxylic acids having 8 to 30 carbon atoms and one carboxylic group per molecule. The alkyl portion has 8 to 30, preferably 12 to 22 carbon atoms. The alkyl portion may be linear or branched, saturated or unsaturated, with linear saturated alkyl preferred. Stearic and myristic acids are preferred carboxylic acids for use in the composition herein. Examples of these carboxylic acids are commercial grades of stearic acid and the like which may contain small amounts of other acids.
- 40 iii. Esters of polyhydric alcohols such as sorbitan esters or glycerol stearate. Sorbitan esters are the condensation products of sorbitol or iso-sorbitol with fatty acids such as stearic acid. Preferred sorbitan esters are monoalkyl. A common example of sorbitan ester is SPAN 60 (ICI) which is a mixture of sorbitan and isosorbide stearates.
- 45 iv. Fatty alcohols, ethoxylated fatty alcohols, alkylphenols, ethoxylated alkylphenols, ethoxylated fatty amines, ethoxylated monoglycerides and ethoxylated diglycerides.
- v. Mineral oils, and polyols such as polyethylene glycol.
- vi. Condensation products of higher fatty acids with polyamines, selected from the group consisting of hydroxyalkyl alkylene diamines and dialkylene triamines and mixtures thereof, as described in US-A-4,661,269.
- 50

- Preferred fabric softeners for use herein are acyclic quaternary ammonium salts, di(hydrogenated)-tallowdimethyl ammonium chloride being most preferred for fabric conditioning compositions of this invention. When the fabric softening composition is used to coat a substrate to form an article according to the present invention the composition includes fabric softeners which can be used singly or in admixture with each other. The fabric softeners are selected from the ammonium salts as mentioned in (1) to (5) above and other softeners as mentioned in (i) to (vi) above.
- 55

About 1% to about 40% of the fabric softening component is used in the compositions of the invention. There must be included at least a sufficient amount of quaternary ammonium salt to achieve anti-static effect, for example about 1% to about 3% in the dilute product and about 2% to about 5% in the concentrated product. On the other hand, the entire fabric softening component may be a quaternary ammonium salt. The diluted version of the product contains about 1% to about 12%, preferably about 3% to about 10% and most preferably about 4% to about 7% of the fabric softening component. The concentrated version of the product contains about 13% to about 40%, preferably about 13% to 30% and most preferably about 13% to about 20% of the fabric softening component.

10 Aminosilicone

The second essential ingredient of the fabric softening composition employed in the present invention is an aminosilicone. Any organosilicone having an amine functionality is suitable for use herein. Aminosilicones employed in the present compositions may be linear, branched or partially crosslinked, preferably linear. Particularly suitable aminosilicones are represented by Formula A:



wherein x and y are numbers of at least 1; a and b are numbers from 1 to 10, preferably from 1 to 5; and R is hydrogen or a hydrocarbon radical, preferably hydrogen. Preferably; x is a number from 50 to 1000 and the ratio of y/(x+y) ranges from 1% to 10%. Typically, aminosilicones having higher amine content exhibit greater compatibility in the mixtures containing an aminosilicone, a Bronsted acid and a fabric softener.

The following list is illustrative of the aminosilicones employed in this invention:

<u>Name</u>	<u>Amine Neutral</u> <u>Milliequivalent</u> <u>gram of silicone</u>	<u>Viscosity</u> <u>(cst)</u>
Magnasoft Fluid ¹	0.5	250
Magnasoft Ultra ¹	0.5	950
SSF ²	0.5	130
CSF ²	0.5	1300
Silicone SL	1.26	350
F-641 ³	0.07	6000
F-751 ³	0.14	500
F-784 ³	0.45	50
F-808 ³	1.6	20

¹Aminosilicone from Union Carbide Corp.

²Aminosilicone from Dow Corning Corp.

³Aminosilicone from Wacker Silicones.

In Silicone SL x=190, y=10, R=hydrogen, a=3 and b=2. Silicone SL is most preferred under current empirical conditions.

Of course, other aminosilicones may be employed.

The aminosilicones included in the compositions herein may be linear, branched, or partially crosslinked, preferably linear, and may range from fluid, liquid to viscous liquid, gum and solid.

The amount of an aminosilicone employed herein typically is about 0.1% to about 20% of the finished composition, and is preferably at least about 0.5% to about 2% to achieve fabric conditioning benefit at an optimum cost, but could be higher in concentrated liquids. When the compositions is used to make articles according to the invention the amount of the amine functional organosilicone is about 3% to about 20% by weight of the composition.

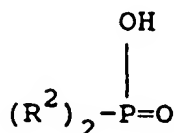
Bronsted Acid

Bronsted acids suitable for use in the present invention contain an alkyl group having at least 6 carbon atoms, preferably 12 to 24 carbon atoms and most preferably 16 to 20 carbon atoms. The alkyl group may be selected from the group of linear or branched alkyl, linear or branched alkenyl, linear or branched alkylaryl or alkenylaryl, linear or branched ethoxylated alcohols, or other alkyl groups. The acid groups combined with the above alkyl groups to give suitable Bronsted acids for the present invention include carboxylic, sulfuric, sulfonic, phosphonic, phosphinic, phosphoric and di-alkyl-sulfosuccinic. Bronsted acids employed in the present invention have 1 to 3 acid groups, and preferably have 1 acid group. If the Bronsted acid contains 2 or 3 acid groups per molecule, it is preferred that the acid groups are located structurally close to each other, such as geminally in the case of di-acids or on adjacent carbons. Bronsted acids employed in the present invention may also be substituted with electron-withdrawing groups such as, for example, a hydroxy group. Examples of Bronsted acids suitable for the present invention include but are not limited to:

- i. C₁₇H₃₅COOH or other fatty acids;
- ii. C₁₁H₂₃-C₆H₄-SO₃H or other alkylaryl sulfonic acids;

iii. $C_{14}H_{29}-O-(C_2H_4O)_n-R^1-COOH$ (wherein n is a number from 1 to 25 and R^1 is an alkyl group having 1 to 3 carbon atoms);

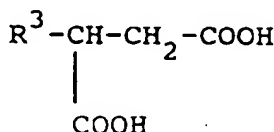
iv.



(wherein R^2 is an alkyl, alkenyl, alkylaryl, alkenylaryl or other alkyl group) or other phosphonic acids;

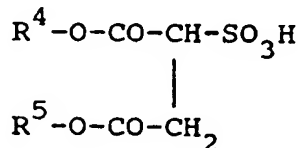
v. $CH_3-(CH_2)_m-PO_3H_2$ (wherein m is a number of at least 5)

vi.



(wherein R^3 is an alkyl, alkenyl, alkylaryl, or other alkyl groups).

vii.



(wherein R^4 and R^5 can be the same or different and can be alkyl, alkenyl, or alkylaryl and may be linear or branched). Preferably both R^4 and R^5 are the same linear alkyl having 6 to 22, most preferably 8 to 18 carbon atoms.

The weight ratio of the Bronsted acid to the combined weight of the fabric softening component and the aminosilicone is from about 1:100 to about 100:1 but must be at least such that the compatibility among the fabric softening component, the aminosilicone and the Bronsted acid is ensured.

As described above, mixtures defined as compatible herein include mutually soluble as well as mutually stable dispersible mixtures. The Compatibility Test is employed to determine whether the particular amount of Bronsted acid compatibilises an aminosilicone with a fabric softening component.

The Compatibility Test is conducted as follows: a 10 gram sample containing a fabric softening component and an aminosilicone is placed into a clear glass flask equipped with a stirring mechanism, such as a magnetic stirrer. A Bronsted acid in the amount of interest is slowly introduced with, conveniently, a Pasteur pipet into the flask, with stirring. If a fabric softening component or an aminosilicone or a Bronsted acid is a solid at room temperature, it is melted before the test is begun with the test taking place above the melting point of the fabric softener or the aminosilicone or the Bronsted acid. Thus, compatibility is defined herein with respect to liquid or liquefied mixtures containing the aminosilicone, the fabric softening component and the Bronsted acid.

If the resulting mixture containing the fabric softening component, the aminosilicone and the Bronsted acid is clear, this indicates that the components of the mixture are mutually soluble and, accordingly, are compatible. Clear mixtures are defined herein as mixtures having about 90% transmittance when measured with visible light probe (one centimeter pathlength) against distilled water background using Brinkman PC800 colorimeter.

The mixture may also become cloudy, indicating that the fabric softening component, the aminosilicone and the Bronsted acid are not mutually soluble at the weight % of the Bronsted acid.

Cloudy samples are placed in an oven at 100°C for at least two hours, then cooled to room temperature and inspected.

Samples which have completely separated into distinct layers are not compatible and are not useful for the invention. Samples which maintain a stable, dispersed or soluble character are compatible and, hence,

useful in the invention. If compatible mixtures solidify on cooling, they may become cloudy, but they remain homogeneous.

Preferably, the components of the mixture containing the fabric softener, the aminosilicone and the Bronsted acid are compatible at a silicone concentration of at least about 2%.

5 Mutually soluble and clear mixtures of the silicone, the fabric softening component and the Bronsted acid indicate the highest degree of compatibility and are preferred. Mutual solubility of the fabric softening component and the aminosilicone is achieved by addition of Bronsted acid.

The amount of Bronsted acid necessary to compatibilise the aminosilicone with the fabric softening component depends on the particular fabric softening component, the aminosilicone and the amounts of the fabric softening component and the aminosilicone used. The appropriate amount of the Bronsted acid is ascertained by the Compatibility Test.

The amount of Bronsted acid needed to compatibilise an aminosilicone with a fabric softening component may be approximated using a calculation based on amine neutral equivalent of the aminosilicone. Amine neutral equivalent (also known as base equivalent) of the aminosilicone is usually indicated on Material Safety Data Sheets obtained from the supplier. Using, for example, DC X2-8122 (an aminosilicone having 1.26×10^{-3} equivalents per gram) and stearic acid (having 3.51×10^{-3} equivalents per gram) the ratio of the aminosilicone to stearic acid is as follows:

$$\begin{array}{r} 3.51 \times 10^{-3} \text{ eq stearic acid/gram stearic acid} \\ \hline 1.25 \times 10^{-3} \text{ eq aminosilicone/gram aminosilicone} \end{array}$$

25 and is equal to 2.79 grams aminosilicone per gram stearic acid. Preferably, a small excess of the Bronsted acid is used.

However, it should be understood that the above calculation based on amine neutral equivalent of the aminosilicone may be used only as a guideline and the mutual compatibility among an aminosilicone, a fabric softener and a Bronsted acid should be ascertained by checking transparency of the mixture containing these components. For example, mutual solubility among the aminosilicone, the fabric softening component and the Bronsted acid also depends on the particular fabric softening component. Where the fabric softening component contains a carboxylic acid the amount of the Bronsted acid necessary to form the mutually soluble mixture may be less than the amount obtained from the above calculation.

35 The fabric conditioning compositions of the invention include a liquid carrier, which is water and which may additionally contain organic solvents such as lower alcohols selected from, for example, methyl alcohol, ethyl alcohol and isopropanol. Both the diluted and the concentrated versions of the product are preferably dispersions of the active ingredients in the water solvent matrix.

The aminosilicone, the fabric softening component and the Bronsted acid which have been ascertained to form a mutually soluble mixture are melted usually at temperatures of less than 100°C for processing convenience and mixed in any order of addition and the resulting mutually soluble mixture is dispersed to form composite particles of the fabric softening component, the aminosilicone and the Bronsted acid in a liquid carrier. Of course, the materials can be spray dried to form discrete softener particles which may also be dispersed in liquid or other forms of product.

45 The composite particles typically form about 1% to about 60% of the fabric conditioning composition of the invention, preferably about 1% to about 30%, and most preferably about 1% to about 20%. Remaining fabric softening component, aminosilicone and the Bronsted acid may be dispersed separately without forming a mutually soluble mixture.

Various additives may be used in combination with the composite particles. These include small amounts of incompatible silicones, such as predominantly linear polydialkylsiloxanes, e.g. polydimethylsiloxanes; alkyl quaternary ammonium salts having one C_8 - C_{30} alkyl chain; soil release polymers such as block copolymers of polyethylene oxide and terephthalate; fatty amines selected from the group consisting of primary fatty amines, secondary fatty amines, tertiary fatty amines and mixtures thereof; amphoteric surfactants; smectite type inorganic clays; anionic soaps; zwitterionic quaternary ammonium compounds and nonionic surfactants.

Other optional ingredients include emulsifiers, electrolytes, optical brighteners or fluorescent agents, buffers, perfumes, colourants, germicides and bactericides.

An article is disclosed for conditioning fabrics in a tumble dryer. The articles of the invention comprises a flexible substrate which carries a fabric conditioning amount of a conditioning composition and is capable of releasing the conditioning composition at dryer operating temperatures. The conditioning composition in turn has a preferred melting (or softening) point of about 25 °C to about 150 °C.

5 The fabric conditioning composition employed in the invention is coated onto a dispensing means which effectively releases the fabric conditioning composition in a tumble dryer. Such dispensing means can be designed for single usage or for multiple uses. One such article comprises a sponge material releasably enclosing enough of the conditioning composition to effectively impart fabric softness during several drying cycles. This multi-use article can be made by filling a porous sponge with the composition. In use, the
10 composition melts and leaches out through the pores of the sponge to soften and condition fabrics. Such a filled sponge can be used to treat several loads of fabrics in conventional dryers, and has the advantage that it can remain in the dryer after use and is not likely to be misplaced or lost.

Another article comprises a cloth or paper bag releasably enclosing the composition and sealed with a hardened plug of the mixture. The action and heat of the dryer opens the bag and releases the composition
15 to perform its softening.

A highly preferred article comprises the compositions containing the softener and the compatible silicone releasably affixed to a flexible substrate such as a sheet of paper or woven or nonwoven cloth substrate. When such an article is placed in an automatic laundry dryer, the heat, moisture, distribution forces and tumbling action of the dryer removes the composition from the substrate and deposits it on the
20 fabrics.

The sheet conformation has several advantages. For example, effective amounts of the compositions for use in conventional dryers can be easily absorbed onto and into the sheet substrate by a simple dipping or padding process. Thus, the end user need not measure the amount of the composition necessary to obtain fabric softness and other benefits. Additionally, the flat configuration of the sheet provides a large surface
25 area which results in efficient release and distribution of the materials onto fabrics by the tumbling action of the dryer.

The substrates used in the articles can have a dense, or more preferably, open or porous structure. Examples of suitable materials which can be used as substrates herein include paper, woven cloth, and non-woven cloth. The term 'cloth' herein means a woven or non-woven substrate for the articles of
30 manufacture, as distinguished from the term 'fabric' which encompasses the clothing fabrics being dried in an automatic dryer.

It is known that most substances are able to absorb a liquid substance to some degree; however, the term 'absorbent', as used herein, is intended to mean a substrate with an absorbent capacity (i.e., a parameter representing a substrate's ability to take up and retain a liquid) from 4 to 12, preferably 5 to 7
35 times its weight of water.

If the substrate is a foamed plastics material, the absorbent capacity is preferably in the range of 15 to 22, but some special foams can have an absorbent capacity in the range from 4 to 12.

Determination of absorbent capacity values is made by using the capacity testing procedures described in U.S. Federal Specifications (UU-T-595b), modified as follows:

- 40 1. tap water is used instead of distilled water;
2. the specimen is immersed for 30 seconds instead of 3 minutes;
3. draining time is 15 seconds instead of 1 minute; and
4. the specimen is immediately weighed on a torsion balance having a pan with turned-up edges.

Absorbent capacity values are then calculated in accordance with the formula given in said Specification. Based on this test, one-ply, dense bleached paper (e.g., Kraft or bond having a basis weight of about
45 14.5 kg (32 pounds) per 3,000 square feet) has an absorbent capacity of 3.5 to 4; commercially available household one-ply towelling paper has a value of 5 to 6; and commercially available two-ply household towelling paper has a value of 7 to about 9.5.

Suitable materials which can be used as a substrate in the invention herein include, among others,
50 sponges, paper and woven and non-woven cloth, all having the necessary absorbency requirements defined above.

The preferred non-woven cloth substrates can generally be defined as adhesively bonded fibrous or filamentous products having a web or carded fibre structure (where the fibre strength is suitable to allow carding), or comprising fibrous mats in which the fibres or filaments are distributed haphazardly or in
55 random array (i.e. an array of fibres in a carded web wherein partial orientation of the fibres is frequently present, as well as a completely haphazard distributional orientation), or substantially aligned. The fibres or filaments can be natural (e.g. wool, silk, jute, hemp, cotton, linen, sisal or ramie) or synthetic (e.g. rayon, cellulose ester, polyvinyl derivatives, polyolefins, polyamides or polyesters).

The preferred absorbent properties are particularly easy to obtain with non-woven cloths and are provided merely by building up the thickness of the cloth, i.e., by superimposing a plurality of carded webs or mats to a thickness adequate to obtain the necessary absorbent properties, or by allowing a sufficient thickness of the fibres to deposit on the screen. Any diameter or denier of the fibre (generally up to about 10 denier) can be used, inasmuch as it is the free space between each fibre that makes the thickness of the cloth directly related to the absorbent capacity of the cloth, and which, further, makes the non-woven cloth especially suitable for impregnation with a composition by means of intersectional or capillary action. Thus, any thickness necessary to obtain the required absorbent capacity can be used.

When the substrate for the composition is a non-woven cloth made from fibres deposited haphazardly or in random array on the screen, the articles exhibit excellent strength in all directions and are not prone to tear or separate when used in the automatic clothes dryer.

Preferably, the non-woven cloth is water-laid or air-laid and is made from cellulosic fibres, particularly from regenerated cellulose or rayon. Such non-woven cloth can be lubricated with any standard textile lubricant. Preferably, the fibres are from 5mm to 50mm in length and are from 1.5 to 5 denier. Preferably, the fibres are at least partially oriented haphazardly, and are adhesively bonded together with a hydrophobic or substantially hydrophobic binder-resin. Preferably, the cloth comprises about 70% fibre and 30% binder resin polymer by weight and has a basis weight of from about 18 to 45g per square meter.

In applying the fabric conditioning composition to the absorbent substrate, the amount impregnated into and/or coated onto the absorbent substrate is conveniently in the weight ratio range of from about 10:1 to 0.5:1 based on the ratio of total conditioning composition to dry, untreated substrate (fibre plus binder). Preferably, the amount of the conditioning composition ranges from about 5:1 to about 1:1, most preferably from about 3:1 to 1:1, by weight of the dry, untreated substrate.

According to one preferred embodiment of the invention, the dryer sheet substrate is coated by being passed over a rotogravure applicator roll. In its passage over this roll, the sheet is coated with a thin, uniform layer of molten fabric softening composition contained in a rectangular pan at a level of about 13.7/m² (15g/square yard). Passage of the substrate over a cooling roll then solidifies the molten softening composition to a solid. This type of application is used to obtain a uniform homogeneous coating across the sheet.

Following application of the liquefied composition, the articles are held at room temperature until the composition substantially solidifies. The resulting dry articles, prepared at the composition substrate ratios set forth above, remain flexible; the sheet articles are suitable for packaging in rolls. The sheet articles can optionally be slitted or punched to provide a non-blocking aspect at any convenient time if desired during the manufacture process.

The amount of the fabric softening composition on the sheet is subject to normal coating parameters such as, for example, viscosity and melting point of the fabric softening component and is typically about 0.5 grams to about 5 grams, preferably about 1 gram to about 3.5 grams. The fabric softening composition employed in the present invention contains 0.1% to about 95% of the fabric softening component. Preferably from about 10% to about 80% and most preferably from about 30% to about 70% of the fabric softening component is employed herein to obtain optimum softening at minimum cost. When the fabric softening component includes a quaternary ammonium salt, the salt is used in the amount of about 10% to about 80%, preferably about 30% to about 70%.

The fabric conditioning compositions of the invention can be used in the rinse cycle of a conventional home laundry operation. Generally, rinse water has a temperature of from about 5°C to about 70°C. The concentration of the total active ingredients is generally from about 2 ppm to about 1000 ppm, preferably from about 10 ppm to about 500 ppm, by weight of the aqueous rinsing bath. When multiple rinses are used, the fabric conditioning compositions are preferably added to the final rinse.

The following Examples will more fully illustrate the embodiments of this invention. All parts, percentages and proportions referred to herein and in the appended claims are by weight of the composition unless otherwise indicated.

Example I

Compatibilising effect of stearic acid in fabric softening mixture containing an aminosilicone and fabric softeners was investigated.

Compatibilised aminosilicone was prepared by blending 3.7 grams of the aminosilicate (Silicone SL) with 1.3 grams of molten stearic acid (Hydrofol Acid 1895). Various fabric softener actives as indicated in Table I were then added to the resulting compatibilised silicone with stirring and sufficient heat to melt all the components. Results that were generated are summarised in Table I.

Table I

5	<u>Fabric Softener</u>	<u>Mutual Solubility of Fabric Softener With</u>	
10		<u>Silicone SL</u>	<u>Compatibilised Silicone SL</u>
15	Mineral Oil (Fisher)	no	yes
	Adogen 442 ¹	no	yes

Adogen 442 = dihydrogenated tallow dimethylammonium
chloride from Sherex Corp.

Adogen 442 was mutually soluble with compatibilised Silicone SL (Silicone SL mixed with stearic acid) up to about 30% of Adogen 442.

Changing the order of addition did not influence the compatibilising effect of stearic acid.

This example demonstrates that addition of stearic acid, which is a Bronsted acid within the scope of the invention compatibilises aminosilicones with various fabric softeners.

Example II

Compatibilising effect of stearic acid in fabric softening mixtures containing an aminosilicone and quaternary ammonium salts was studied. The amount of stearic acid necessary to compatibilise an aminosilicone with a fabric softening agents approximated using an amine neutral equivalent.

The results that here obtained are summarised in Table I.

Table I

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<u>Components</u>	<u>Weight (grams)</u>			
<u>Sample</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Aminosilicone	0.2	2.5	0.1	2.5
Adogen 343 ¹	10	10	-	-
Varisoft 137 ²	-	-	10	10
Stearic acid	-	0.89	-	0.89

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¹Adogen 343 = dihydrogenated tallow methyl amine from Sherex Corp.

²Verisoft 137 = dihydrogenated tallow dimethyl ammonium methylsulfate from Sherex Corp.

Observations:

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Samples 1 and 3 were incompatible as determined by the Compatibility Test at aminosilicone concentration of 2% and 1% respectively. Samples 2 and 4 were compatible as determined by the Compatibility Test at 25% silicone concentration.

This example demonstrates that stearic acid, a Bronsted acid within the scope of the invention, compatibilises aminosilicones with fabric softening agents as determined by the Compatibility Test.

Example III

Example IIIA:

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3g of dihydrogenated tallow dimethylammonium chloride (Adogen 442) was added to 5g mineral oil, 3.7g Silicone SL, and 1.3g Hydrofol Acid 1895 (stearic acid) with stirring and heating. The resulting mixture was a clear, homogeneous, one-phase system.

Example IIIB:

A 5% aqueous dispersion of composition particles containing the mutually soluble mixture of Example IIIA was then prepared by adding the mutually soluble mixture to water, with heating to liquify the solids, and stirring with an overhead stirrer. Stirring was maintained while the dispersion was cooled to room temperature. Additional particle size reduction was achieved by passing it through a Gaulin homogeniser at 6000 PSI.

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Example IIIC:

A series of fabric conditioning formulations was prepared by blending the dispersion prepared in Example IIIB with 35% emulsion of Silicone SL containing 2% Adogen 442 based on silicone weight. The samples in this series contained 5%, 10%, 20%, 30%, 40% and 50% of the dispersion of Example IIIB.

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This example demonstrates that addition of stearic acid, which is a Bronsted acid within the scope of the invention, compatibilises an aminosilicone with a cationic fabric softener.

The example further illustrates that a liquid fabric softening composition containing composite particles of the mutually soluble mixture of the aminosilicone, the fabric softener and the Bronsted acid within the scope of the invention can be prepared and can be incorporated with other fabric treating ingredients.

Example IV

Fabric softening formulations incorporating composite particles of the invention were prepared as summarised in Table II. Samples A, B, C and D contain composite particles of aminosilicone (Silicone SL), stearic acid, mineral oil and Adogen 442.

Sample B is a dispersion prepared in the same manner as sample A, but contains 10.0g of mineral oil instead of 5g of mineral oil in sample A.

Sample C is a 1:1 blend of sample A with 5% dispersion of Adogen 442, also containing 15 ppm NaCl.

Sample D is a 1:1 blend of sample A with 5% dispersion of Varisoft 445 (methyl-1-hydrogenated tallow amidoethyl-2-hydrogenated tallow imidazolinium methyl sulfate from Sherex Corp.), also containing 15 ppm CaCl_2 .

Table II

Liquid Fabric Conditioning Rinse Compositions

<u>Ingredients</u>	<u>Sample</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Adogen 442		1.15	0.83	2.92	0.42
Varisoft 445		-	-	-	2.50
Mineral Oil		1.92	2.78	1.39	1.39
Silicone SL		1.41	1.02	0.50	0.50
Stearic Acid		0.52	0.37	0.19	0.19
NaCl		-	-	tr.	-
CaCl_2		-	-	-	tr.
Perfume		-	-	tr.	tr.
Colourant		-	-	tr.	tr.
Preservative		-	-	tr.	tr.
Water		-----to 100%-----			

Samples A, B, C and D from Table II were evaluated for their softening properties. Terry cloths were prewashed with a solution of Neodol 25-9 (ethoxylated alcohol from Shell Corp.) and Na_2CO_3 to remove textile finishes on the surface, rinsed with the samples in a Tergometer and then line dried. The cloth load was 35 g per litre of water. The concentration of composite particles was 0.1 g per litre of water. The concentration of composite particles was 0.1 g per litre of water. The control samples were rinsed in water only. For both the experimental and control samples, water hardness of 120 ppm was used. Using paired comparisons, a panel of 20 judges assessed the softness of the treated cloths vs. controls. All panellists preferred the treated cloths over the controls in all evaluations.

Example V

The ability of Bronsted acids to compatibilise amine-functional silicones with quaternary ammonium salts was investigated.

Example VA:

10g of a fabric softener which is a mixture of dihydrogenated tallow-di-methyl ammonium methylsulfate (70%) and C₁₄-C₁₈ fatty acids (30%) was placed in a small vial and melted with stirring. Silicone SL (an aminosilicone) was added to produce a mixture which is 25% silicone by weight (3.33g of silicone). The resulting mixture was opaque but stable as determined by the Compatibility Test.

Based on amine neutral equivalent calculation, the amount of stearic acid necessary to compatibilise 3.3g of DC X2-8122 is 1.2g. However, the fabric softener already contained 3g of fatty acids. Thus, it was not necessary to admix additional Bronsted acid to attain the compatible mixture of Example VA.

The example demonstrates that the amount of Bronsted acid necessary to compatibilise an aminosilicone with a fabric softening component must be ascertained using the Compatibility Test and the amount based on amine neutral equivalent calculation can be used only as a guideline.

Example VI

Compatible fabric conditioning mixtures were prepared. Di(hydrogenated tallow)-di-methyl ammonium methyl sulfate (Varisoft 137 from Sherex Corp.) was combined with commercially available aminosilicones and other softeners in various proportions as indicated in Table II.

The mixtures were all found to be homogeneous and stable at processing and use temperatures.

Table IIWeight percent of formulation

<u>Code</u>	<u>Varisoft 137</u>	<u>Stearic Acid</u>	<u>Silicone</u>	<u>Magnasoft</u>	<u>Span</u>
			<u>SL</u>	<u>Ultra</u>	<u>60</u>
A	70	10	-	20	-
B	70	10	2	-	-
C	23	7	20	-	50
D	70	20	-	10	-
E	70	20	10	-	-

Formulation E from Table II above was fabricated into an article for use in the tumble dryer by coating the molten composition onto sheets of spun bonded polyester using a two roll coating machine.

The article with the solidified softening composition was placed in a tumble dryer with freshly laundered clothing and the dryer was operated in the normal fashion for one hour. Upon removal, the clothing was judged to have excellent antistatic properties. The weight loss of the softening article was assessed and it was judged that the softening composition transferred to the clothing in the environment of the dryer. A 20 member employee panel then judged the clothing to have superior softness when compared to control samples without softener in a pair comparison test.

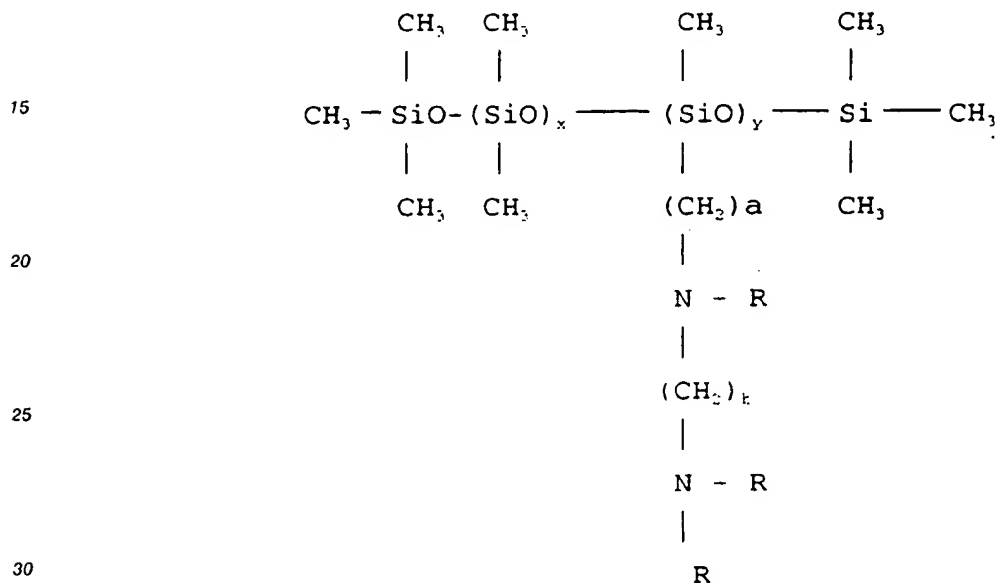
Claims

- Discrete composite particles characterised in that they consist of a mutually compatible mixture of:
 - at least 1 wt% of a fabric softening component comprising a cationic quaternary ammonium salt,

b) an amine functional organosilicone,
 c) a Bronsted acid containing an alkyl group having at least 6 carbon atoms,
 the weight ratio of (c) : [(a) + (b)] being such that the mixture when in liquefied form is homogeneous.

- 5 2. Particles according to claim 1, characterised in that they consist of a mutually soluble mixture of (a), (b) and (c) which when in liquefied form; is a clear homogeneous liquid.
3. The particles as claimed in claim 1 or claim 2 characterised in that the structure of the amine functional organosilicone (b) is

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wherein x and y are numbers a of at least 1; a and b are numbers from 1 to 10; and R is hydrogen or a hydrocarbon radical.

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4. Particles of any preceding claim characterised in that the Bronsted acid (c) is a carboxylic, sulphuric, sulphonic, phosphonic, phosphinic, phosphoric or di-alkylsulphosuccinic acid.
5. Particles as claimed in any preceding claim, characterised in that the Bronsted acid (c) is stearic acid.
- 40 6. Particles as claimed in any preceding claim, characterised in that the cationic quaternary ammonium salt is selected from acyclic quaternary ammonium salts having at least two C₈₋₃₀ alkyl chains, quaternary imidazolinium salts, diamido quaternary ammonium salts, biodegradable quaternary ammonium salts and mixtures thereof.
- 45 7. Particles as claimed in any preceding claim characterised in that the quaternary ammonium salt is selected from dihydrogenatedtallowdimethyl ammonium chloride and ditallowimidazolinium chloride.
8. A liquid fabric conditioning composition characterised in that it comprises from 1 to 60 wt% of the particles as claimed in any of the preceding claims.
- 50 9. A liquid fabric conditioning composition according to claim 8, comprising composite particles of a mutually compatible mixture of:
- (a) 1% to 40% by weight of the composition of a fabric softening component comprising a cationic quaternary ammonium salt;
- 55 (b) 0.1% to 20% by weight of said composition of an amine functional organosilicone; and
- (c) a Bronsted acid having at least 6 carbon atoms,
 the weight ratio of (c) : [(a) + (b)] being such that the mixture when in liquefied form is homogeneous.

10. A composition as claimed in claim 9, characterised in that the amine functional organosilicone (b) is present in an amount of from 0.5% to 20% by weight of the composition.

11. An article for conditioning fabrics, comprising a flexible substrate carrying thereon particles as claimed in any one of claims 1 to 7.

12. An article as claimed in claim 11, characterised in that the particles carried on the substrate contain from 0.1% to 20% of the amine functional organosilicone (b).

13. An article as claimed in claim 12, characterised in that the particles carried on the substrate contain from 3% to 20% by weight of the amine functional organosilicone.

14. An article as claimed in any one of claims 11 to 13, characterised in that the flexible substrate is in a sheet configuration.

Patentansprüche

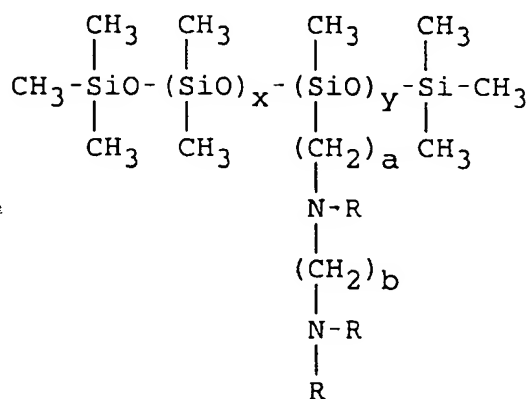
1. Einzelne Verbundteilchen, dadurch gekennzeichnet, daß sie aus einem gegenseitig verträglichen Gemisch von:

- a) mindestens 1 Gew.-% einer textilweichmachenden Komponente, umfassend ein kationisches quaternäres Ammoniumsalz,
- b) einem aminfunktionellen Organosilicon,
- c) einer Brönstedsäure, enthaltend eine Alkylgruppe mit mindestens 6 Kohlenstoffatomen, bestehen, wobei

das Gewichtsverhältnis (c) : [(a) + (b)] derart ausgelegt ist, daß das Gemisch in verflüssigter Form homogen ist.

2. Teilchen nach Anspruch 1, dadurch gekennzeichnet, daß sie aus einem gegenseitig löslichen Gemisch von (a), (b) und (c) bestehen, das in verflüssigter Form eine klare, homogene Flüssigkeit ist.

3. Teilchen nach Anspruch 1 oder Anspruch 2, dadurch gekennzeichnet, daß die Struktur des aminfunktionellen Organosilicons (b) ist:



worin x und y die Zahlen von mindestens 1 sind; a und b die Zahlen von 1 bis 10 sind und R Wasserstoff oder einen Kohlenwasserstoffrest darstellt.

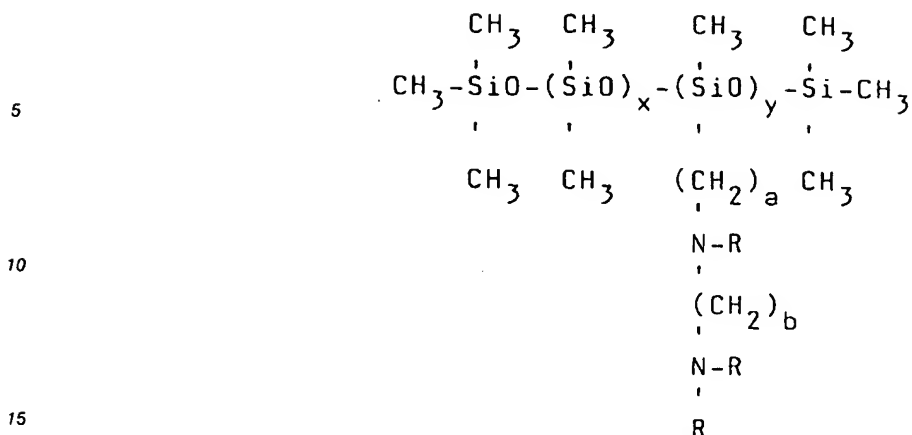
4. Teilchen nach einem vorangehenden Anspruch, dadurch gekennzeichnet, daß die Brönstedsäure (c) eine Carbon-, Schwefel-, Sulfon-, Phosphon-, Phosphin-, Phosphor- oder Dialkylsulfobernsteinsäure ist.

5. Teilchen nach einem vorangehenden Anspruch, dadurch gekennzeichnet, daß die Brönstedsäure (c) Stearinsäure ist.

6. Teilchen nach einem vorangehenden Anspruch, dadurch gekennzeichnet, daß das kationische quaternäre Ammoniumsalz ausgewählt ist aus acyclischen quaternären Ammoniumsalzen mit zumindest zwei C₈₋₃₀-Alkylketten, quaternären Imidazoliniumsalzen, Diamido-quaternären Ammoniumsalzen, bioabbaubaren quaternären Ammoniumsalzen und Gemischen davon.
7. Teilchen nach einem vorangehenden Anspruch, dadurch gekennzeichnet, daß das quaternäre Ammoniumsalz ausgewählt ist aus Di(hydriertem Talg)dimethylammoniumchlorid und Ditalgimidazoliniumchlorid
8. Flüssiges Textilkonditionierungsmittel, dadurch gekennzeichnet, daß es 1 bis 60 Gew.-% der Teilchen nach einem der vorangehenden Ansprüche umfaßt.
9. Flüssiges Textilkonditionierungsmittel nach Anspruch 8, umfassend Verbundteilchen eines gegenseitig verträglichen Gemisches von:
 - (a) 1 bis 40 Gew.-% des Mittels, einer textilweichmachenden Komponente, umfassend ein kationisches quaternäres Ammoniumsalz;
 - (b) 0,1 % bis 20 Gew.-% des Mittels, einem aminfunktionellen Organosilicon und
 - (c) einer Brönstedsäure mit mindestens 6 Kohlenstoffatomen, wobei das Gewichtsverhältnis (c) : [(a) + (b)] derart ausgelegt ist, daß das Gemisch in verflüssigter Form homogen ist.
10. Mittel nach Anspruch 9, dadurch gekennzeichnet, daß das aminfunktionelle Organosilicon (b) in einer Menge von 0,5 bis 20 Gew.-% des Mittels vorliegt.
11. Gegenstand zum Konditionieren von Textilien, umfassend ein biegsames Substrat, das darauf Teilchen nach einem der Ansprüche 1 bis 7 enthält.
12. Gegenstand nach Anspruch 11, dadurch gekennzeichnet, daß die Teilchen, getragen auf dem Substrat, 0,1 bis 20 % von dem aminfunktionellen Organosilicon (b) enthalten.
13. Gegenstand nach Anspruch 12, dadurch gekennzeichnet, daß die auf dem Substrat getragenen Teilchen 3 bis 20 Gew.-% des aminfunktionellen Organosilicons enthalten.
14. Gegenstand nach einem der Ansprüche 11 bis 13, dadurch gekennzeichnet, daß das biegsame Substrat in Folienform vorliegt.

Revendications

1. Particules composites séparées, caractérisées en ce qu'elles consistent en un mélange mutuellement compatible de :
 - a) au moins 1% en poids d'un composant d'adoucissement des textiles, comprenant un sel d'ammonium quaternaire cationique,
 - b) une organosilicone aminofonctionnelle,
 - c) un acide de Bronsted contenant un radical alkyle ayant au moins 6 atomes de carbone,le rapport pondéral de (c) : ((a) + (b)) étant tel que le mélange est homogène quand il est sous forme liquéfiée.
2. Particules selon la revendication 1, caractérisées en ce qu'elles consistent en un mélange mutuellement soluble de (a), (b) et (c) qui est un liquide homogène limpide quand il est sous forme liquéfiée.
3. Particules selon la revendication 1 ou 2 caractérisées en ce que la structure de l'organosilicone aminofonctionnelle (b) est :



dans laquelle x et y sont des nombres d'au moins 1 ; a et b sont des nombres d'au moins 1 à 10 ; R est l'hydrogène ou un radical hydrocarboné.

4. Particules selon l'une quelconque des revendications précédentes caractérisées en ce que l'acide de Bronsted (c) est un acide carboxylique, sulfurique, sulfonique, phosphonique, phosphinique, phosphorique ou di-alkylsulfosuccinique.
5. Particules selon l'une quelconque des revendications précédentes caractérisées en ce que l'acide de Bronsted (c) est l'acide stéarique.
6. Particules selon l'une quelconque des revendications précédentes caractérisées en ce que le sel d'ammonium quaternaire cationique est choisi parmi les sels d'ammonium quaternaire acycliques ayant au moins deux chaînes alkyle en C_8-30 , les sels d'imidazolinium quaternaire, les sels d'ammonium quaternaire diamido, les sels d'ammonium quaternaire biodégradables et leurs mélanges.
7. Particules selon l'une quelconque des revendications précédentes, caractérisées en ce que le sel d'ammonium quaternaire est choisi parmi le chlorure de di-sulf hydrogéné-diméthylammonium et le chlorure de di-sulf-imidazolinium.
8. Composition liquide de conditionnement des textiles, caractérisée en qu'elle comporte de 1 à 60% en poids des particules selon l'une quelconque des revendications précédentes.
9. Composition liquide de conditionnement des, liquides selon la revendication 8, comportant des particules composites d'un mélange mutuellement compatible de :
 - (a) 1 à 40% en poids de la composition d'un composant d'adoucissement des textiles comprenant un sel d'ammonium quaternaire cationique ;
 - (b) 0,1 à 20% en poids de ladite composition d'une organosilicone aminofonctionnelle ; et
 - (c) un acide de Bronsted ayant au moins 6 atomes de carbone,
 le rapport pondéral de (c) : ((a) + (b)) étant tel que le mélange est homogène quand il est sous forme liquéfiée.
10. Composition selon la revendication 9, caractérisée en ce que l'organosilicone aminofonctionnelle (b) est présente en une quantité allant de 0,5 à 20% en poids de la composition.
11. Article pour le conditionnement des textiles, comprenant un substrat souple portant des particules selon l'une quelconque des revendications 1 à 7.
12. Article selon la revendication 11, caractérisé en ce que les particules portées par le substrat contiennent de 0,1 à 20% de l'organosilicone aminofonctionnelle (b).

13. Article selon la revendication 12, caractérisé en ce que les particules portées par le substrat contiennent de 3 à 20% en poids de l'organosilicone aminofonctionnelle.

5 14. Article selon l'une quelconque des revendications 11 à 13, caractérisé en ce que le substrat souple a une configuration en feuille.

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